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MONTHLY PROGRESS REPORT

No. 9

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March 1963 [Osmotic Still and Ionics Dual Membrane Fuel Cell Development]

Submitted by

NEW PRODUCT RESEARCH OF TAPCO

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I INTRODUCTION

This document represents the ninth monthly report covering the work on the experimental program for development of an "Osmotic Still" and improvements in the performance characteristics of the Ionics Dual Membrane Fuel Cell during the month of March 1963. This development work is being accomplished under NASA-Lewis Research Center Contract No. NAS 3-2551 by the New Product Research Department of TAPCO, and Ionics Inc. as a subcontractor to TAPCO.

II OVERALL PROGRESS

A. Tapco Portion of Program

1. The final performance test with the AMF C-60 membrane was completed. A water extraction rate of 136 cc/hr ft^2 was obtained at an electrolyte temperature of 200°F and a vapor pressure of 4.36 psia and an acid pressure of 9.0 psia.

2. A preliminary design of the 2 KW unit has been completed and the design specifications are as follows:

H_2SO_4 Concentration = 6 N or 25%

Average H_2SO_4 Temperature = 200°F

H_2O Extraction Rate = 2 lb/hr = 908 cc/hr

Active Membrane Area = 7 ft^2

Active Membrane Diameter = 9.6"

Number of Membranes = 14

Design Vapor Pressure Differential = approx. 7 psi

Design H_2SO_4 Flow Rate = 0.69 GPM (acid chambers in series)

Design Condenser Coolant Flow Rate = 1.0 GPM

3. The plastic materials compatibility tests were completed. Results of these tests are presented under Section V, Test Results.

4. Preliminary investigation of gasketing materials for the 2 KW unit has been completed. Various methods of application of silicone rubber compounds have been tested. In general, these compounds may be adequate, but application technique is extremely critical. The two compounds used in these tests were General Electric RTV 102 and Dow Corning Selastic RTV 503.

5. Preliminary design of the test rig for the 2 KW still 100 hour performance test is shown schematically in TRW Figure 1A.

B. Ionics Portion of Program

1. Progress was made in long term, high current density and high power density single cell performance and in the assembly and test of multiple-cell batteries.

2. Cells were run continuously up to 1000+ hours with satisfactory performance. They were removed from the test rig to make way for 5-cell batteries.

3. Following a 1000+ hour test, a cell was successfully run at about 150 amps/ft^2. A maximum power density of over 40 watts/ft^2 was achieved.

4. The test rigs were modified to accept 5-cell batteries. Techniques to assemble and fill the batteries were developed.

5. Two 5-cell batteries were assembled and run successfully for 100 hours each, one at 60°C and one at 30°C. Output voltages were over 3.5V at 24 amps/ft^2, compared to the 3.4V at this current density needed to parallel the requirements of the ten-cell battery.

III CURRENT PROBLEMS

A. Tapco Portion of Program

There have been no major technical problems during this reporting period.

B. Ionics Portion of Program

1. Data obtained on 5-cell batteries must be fully evaluated.

2. Choice of components for the 10-cell Demonstrator Battery must be finalized.

IV NEXT MONTH'S EFFORTS

A. Tapco Portion of Program

1. Finalize 2KW unit design.

2. Complete detail drawings of 2KW unit.

3. Finalize test rig design.

4. Procure all raw material and parts for test unit and test rig.

5. Fabricate 2KW unit.

6. Initiate test rig assembly.

B. Ionics Portion of Program

1. Complete tests on five 5-cell batteries.

2. Continue data evaluation of 5-cell battery tests.

3. Finalize choice of components for 10-cell Demonstrator Battery, fabricate components and complete initial assembly of the battery.

V. TEST RESULTS

A. Tapco Portion of Program

1. The results of a 30 day test on polyvinyl dichloride and polymethyl methacrylate material after being exposed to a 30% solution of sulfuric acid at 200°F in a reflux condenser are expressed in TRW Table 1A.

As a result of these tests, the polyvinyl dichloride material has been selected as the material of construction for the acid and vapor chambers of the 2KW osmotic still.

B. Ionics Portion of Program

1. Single Cell Tests

The two, 36 sq. in. test cells recorded as still running last month (Cells D9228 and E1651) were continued on test until the test stands were needed for use with 5-cell batteries. In addition, two other single cells (E1679 and E1685) were constructed, tested and disassembled. One of these (E1679) was used to follow up an apparent lead uncovered in prior 4 sq. in. cell work; the other (E1685) was assembled from the same components that went into the first 5-cell battery (Battery 1). Detailed data are given in Table 1.

1.1 Successful Long-Term Runs

Cell D9228 was run for over 500 hours and Cell E1651 for over 1000 hours while meeting electrical requirements. They were removed from the test rigs to make room for 5-cell batteries. Both cells tended to improve somewhat with time. The measured specific conductance increased again after an intermediate low value.

	SPECIFIC CONDUCTANCE Mhos/ft^2	
	D9228	E1651
Initial	158	266
Intermediate Low	98	121
Final	178	226

1.2 High Current and Power-Density Tests

Cell E1651 was successfully run at a current density of about 150 amps/ft^2 for a 22-hour period. The cell output voltage decreased somewhat with time, 0.21V initially, 0.19V finally. The test showed that high current densities can be maintained by Dual Membrane fuel cells.

A polarization curve was obtained in the 0-100 amps/ft^2 region. The total potential between the cell terminals was determined at various current densities. In addition, a standard calomel reference electrode was immersed in the electrolyte stream leaving the cell. The potential between each cell terminal and the reference electrode was also determined at each current density. A schematic of the hook-up is shown in Figure 1. The data obtained are shown in Figure 2.

A curve of power-density (watts/ft^2) vs current density was prepared from these data and is presented in Figure 3. A point, representing "average" conditions of the 150 amps/ft^2 run, is included. Power densities of over 40 watts/ft^2 were obtained.

1.3 High-Teflon Oxygen Electrode

The Sixth Monthly Report (February 10, 1963) included results of experiments carried out in 4 sq. in. cells where the amount of Teflon used in preparing electrodes was varied. Optimum results were obtained when the oxygen electrode was made with a paste including 4 times as much Teflon dispersion as normal and the hydrogen electrode had only 1/2 the normal amount.

To follow up this lead, Cell E1679 was assembled with an oxygen electrode that had four times the normal amount of Teflon. A standard hydrogen electrode was used as attempts to prepare one using only 1/2 as much Teflon dispersion were unsuccessful: the paste did not stick well to the platinum gauze.

Unfortunately, this cell did not perform at all well and the multiple-cell batteries will be assembled using standard electrodes.

1.4 Comparison Cell

A single 36 sq. in. cell (E1685) was assembled of the same components used in Battery 1. (See Table 2.) Gas pressure drop data were obtained to provide a check on the operation of Battery 1. The data are included in Section 2 which deals with 5-cell batteries.

2.0 5-Cell Batteries

The test rig was modified to permit handling several 5-cell batteries simultaneously. Techniques for assembling and filling the batteries were tested and finalized. Two 5-cell batteries were assembled during the month of March, 1963 and subjected to 100-hour performance tests

at the prescribed current density of 24 amps/sq. ft. The effect of varying some of the flow parameters was also investigated.

2.1 Assembling and Filling 5-Cell Batteries

Forms and systems were devised and employed to provide multiple quality assurance checks during the assembly of the batteries. All compartments and electrodes were individually identified and their correct emplacement with regard to location, direction and right or left handedness within the battery stack was verified and recorded.

The electrolyte compartments were filled by sucking up 6N H_2SO_4 through the acid inlet manifold with the aid of a slight vacuum (13.2 psia) applied to the acid exit manifold. The filling rate was adjusted by means of a variable constriction in the acid inlet line to about 20 ml/min. Prior tests with a dummy electrolyte compartment faced with a lucite cover plate showed that this technique would result in bubble-free filling of the electrolyte compartments.

The work with this dummy compartment also suggested a modification in the design of the top (exit end) of electrolyte compartments. A slight "cathedral ceiling" (mid-point raised 3/16" above the horizontal) would facilitate the removal of any gas bubbles that might otherwise remain in the compartment. Such a modification in the design of electrolyte compartments has been adopted and implemented.

After filling a battery, the inlet acid manifold was closed and the vacuum on the electrolyte compartments maintained for some time in order to check that gas would not leak into the electrolyte compartments through possible defects in the membranes or through cross-leaks in the manifold system or through the side walls of the cell assembly. If gas bubbles appeared in the electrolyte exit manifold, or if electrolyte would flow out of this manifold (indicating internal gas bubble appearance), suitable steps were taken to close off the particular leak in question. All batteries were "bubble-tight" prior to installation on the test rig.

2.2 Characteristics of Batteries 1 and 2

A summary of the components, operating conditions, and performance of Batteries 1 and 2 is given in Table 2. Detailed log plots of voltage, liquid accumulation rates, and pressure drops are given in Figures 4 through 14.

Both batteries used Teflon compartments and tantalum pusher and collector plates. However, the electrolyte compartments of Battery 1 were of thicker stock than those of Battery 2 (roughly 1/8" vs 1/10") and contained two layers of Trilok instead of 1. The main difference between the two batteries was that Battery 1 was tested at 60°C and Battery 2 at 30°C.

Battery 1 performed satisfactorily from the start. A current density of 24 amps/ft^2 was maintained at better than 3.5 volts throughout the 165 hours that the battery was on test. This may be compared with the 3.4 volts for a 5-cell battery that corresponds to the average cell voltage of 0.68 volts required for the 10-cell Demonstrator Battery.

During the first 113 hours, Battery 1 was run with gas rates of about 12 times the gas consumption rates - flow rates of approximately 2 liters/min. of H_2, 1 l/m of O_2 - and a fairly high electrolyte rate of 60 ml/min. (Run 1-A). Thereafter, the gas rates were cut down to about 3 times the consumption rate (Run 1-C). For both 1-B and 1-C, the electrolyte rate was reduced to 7 ml/min.

Operation at the low gas rate (1-B) was not stable: every 8 hours or so the voltage developed in one cell would start to drop, apparently due to liquid accumulation in the gas compartment. A sudden short decrease in downstream pressure would result in a rapid discharge of liquid and return of the cell voltage to normal. At the high and intermediate rates (1-A and 1-C), no such instability occurred, apparently the more significant gas compartment pressure drops (60 and 30 mm H_2O vs 14 mm H_2O in 1-B) were sufficient to ensure adequate purging of liquid from the gas compartments.

The voltages developed showed no trend with time. They were somewhat higher for 1-B and 1-C than for 1-A. This may be due to the higher internal temperature resulting from a decrease in electrolyte flow rate.

The pressure drops measured across the compartments were more or less in line with those obtained in the single cell made of some of the same components (Cell E1685).

The electrolyte pump operation was such that the pump would operate in either one of two modes: it would either operate continuously giving a fairly constant pressure drop through the battery, or it would pump intermittently, being controlled by high and low pressure relays. This second mode of operation would give a pressure drop cycling in level between high (pump pumping) and low (pump not pumping). The resulting data are shown in Figure 8.

Battery 2 did not give quite as satisfactory performance. Initially, fairly stable operation could only be obtained by running the electrolyte compartments at a slightly higher pressure than the gas compartments (about 5 psig vs 2.3 psig). Even so, a slight deterioration in voltage with time was observed. After 48 hours of this sort of operation (Run 2-A-1) performance became erratic for 4 hours but was brought back to an acceptable level by eliminating the deliberate pressure

imbalance. The second half of Run 2A (Run 2-A-2) was then completed. Removal of the pressure imbalance also brought the liquid accumulation rates down appreciably. The initial requirement for a pressure imbalance suggests that contact between electrodes and membranes was not as good as it should have been. However, Battery 2 did complete 100 hours at 30°C and a current density of 24 amps/ft^2 while developing a voltage averaging over 3.5 volts. Following Run 2-A, the temperature of the bath surrounding Battery 2 was raised to 60°C and another 100+ hours run completed. This will be reported in the tenth monthly report covering work performed in April 1963.

VI QUALITY ASSURANCE

A. Tapco Portion of Program

The Quality Assurance Status of the program at both TAPCO and Ionics is considered to be satisfactory as it relates to the design, fabrication, and test activity carried out during this reporting period.

TABLE I

Single 36 Sq. In. Cell Tests

Cell No.	Cell Components	Temp. °C.	Press. PSIG	Amps	Voltage	Time Hours	Spec. Cond. Mhos/Ft.^{2}	Remarks
D 9228	Sintered (Teflon) Electrodes Std.Memb. Tantalum Pusher & Collector Plates	Room Temp.	5	4	0.728	264	102	
					0.760	360	187	
					0.752	384	184	
					0.740	432	140	
					0.758	456	135	
					0.740	480	133	
					0.758	504	147	
					0.760	528	178	
				4		554		
				16	0.36	554		
				12	0.52	554		
				12		603		
				4	0.742	603		
				4	0.750	627		Removed from rig
E 1651	Sintered (Teflon) Electrodes Std. Memb. Tantalum Pusher & Collector Plates	60	5	4	0.782	504	121	
					0.782	552		
					0.808	600		
					0.735	672	200	
					0.828	744		
					0.825	840	167	
					0.830	888		
					0.818	1008		
				4	0.815	1032	226	
				6	0.775	1032		
				6	0.778	1080		
				0	1.04	1080		
				0	1.05	1104		
				41	0.21	1104		
				36	0.19	1126		
				4	0.81	1126		
				4	0.81	1152		Removed from rig

TABLE I (Contd.)

Single 36 Sq. In. Cell Tests

Cell No.	Components	Temp. °C.	Press. PSIG	Amps	Voltage	Time Hours	Spec. Cond. Mhos/Ft.^{2}	Remarks
E 1679	O_2 Electrode had 4 times regular amt teflon. H_2 electrode: standard sintered. Std. Memb. Gold plated Titanium Pusher Plates Epoxy Comp. Tantalum Collector Plates	60	5	4	0.632 0.635	5 51	102	Discontinued because of poor performance
E 1685	As Battery 1 See Table 2	60	5	4 6	0.782 0.70	16 16	95	Removed from rig

TRW TABLE - 1A

Plastic Material Compatibility Test With 30% H_2SO_4 at 200°F

Sample Description	Dimensions (Inches) Before	Dimensions (Inches) After	Hardness (Barcol) Before	Hardness (Barcol) After	Weight (Grams) Before	Weight (Grams) After	Remarks
Polymethyl Methacrylate							
Uncertified Sample A-1	0.9975 x 1.002 x 0.247	0.999 x 1.025 x 0.244	49	42	4.6773	4.7197	Thickness ranged from 0.241 to 0.246
Uncertified Sample A-2	0.9980 x 1.003 x 0.247	0.984 x 1.040 x 0.241	49	42	4.6799	4.7233	Thickness ranged from 0.239 to 0.243
Certified Sample B-1	0.999 x 1.002 x 0.226	0.999 x 1.0025 x 0.226	50	42	4.3550	4.3904	
Certified Dample B-2	0.999 x 1.002 x 0.224	1.000 x 1.002 x 0.225	49	41	4.3292	4.3628	
Polyvinyl Dichloride							
Certified Sample C-1	0.999 x 1.005 x 0.126	0.999 x 1.005 x 0.127	3	4	3.1662	3.1855	
Certified Sample C-2	0.998 x 1.005 x 0.126	0.998 x 1.002 x 0.127	5	6	3.1593	3.1765	
Uncertified Sample D-1	0.983 x 0.996 x 0.123	0.990 x 0.986 x 0.121	5	5	2.9733	2.9920	Thickness ranged from 0.119 to 0.123
Uncertified Sample D-2	0.984 x 1.004 x 0.123	0.989 x 1.004 x 0.121	8	8	3.0026	3.0191	Thickness ranged from 0.121 to 0.126

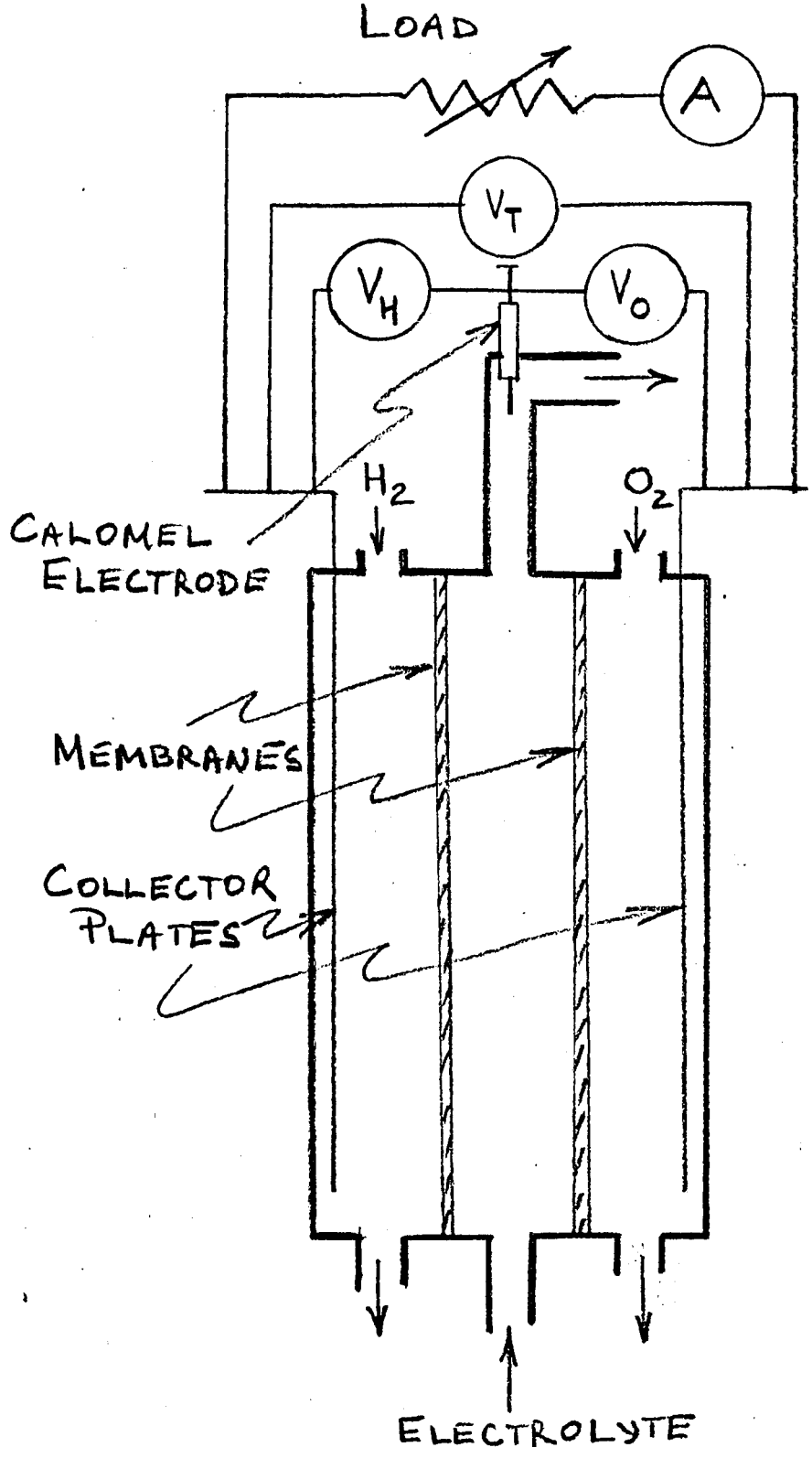
TABLE 2

SUMMARY OF BATTERY OPERATION

	BATTERY 1			BATTERY 2	
Components					
Endplates	Stainless Steel				
Insulators	Butyl Rubber				
Collector Plates	10 mil. Tantalum				
Gas Compartments	64-65 mil. Teflon				
Pusher Plates	10 mil. Tantalum				
Electrodes	Standard Sintered				
Membranes	IONICS 61-AZG				
Electrolyte Comp.	118-133 mil. Teflon			99-102 mil. Teflon	
Trilok Fillers	2 pcs/cell (#6027-1-1)			1 pc/cell (#6027-1-1)	
Gaskets & Grommets	8 mil. dacron-backed Viton				
Operating Conditions Run No.	1-A	1-B*	1-C	2-A-1	2-A-2
Rates					
H_2, liters/min	2.10	0.55	0.90	2.2	2.2
O_2, liters/min	1.02	0.21	0.44	1.05	1.05
Electrolyte, ml/min	60	7	7	60	60
Pressures					
H_2, psig.	5.0	5.0	5.0	2.5	3.0
O_2, psig.	5.0	5.0	5.0	2.1	3.2
Electrolyte, psig.	4.7-5.1	4.7-5.1	4.7-5.1	4.7-5.1	3.0
Current Load, A/Ft^2	24	24	24	24	24
Temperature, ^oC	60	60	60	30	30
Duration, Hours	112	24	27	48	52
Performance					
Pressure Drops					
H_2, mm. H_2O	46	15	25	50	50
O_2, mm. H_2O	60	14	30	100	100
Liquid Accumulation					
H_2 side: H_2O, gm/hr	6.6	5.5	7.6	4.8	1.35
H_2SO_4, gm/hr	1.4	1.6	0.95	1.4	0.26
O_2 side: H_2O, gm/hr	5.2	4.3	7.5	7.5	2.6
H_2SO_4, gm/hr	1.1	1.1	1.25	1.8	0.47
Voltages @ $24A/Ft^2$					
Cell 1	0.670	0.684	0.684	.670	.678
Cell 2	0.745	0.760	0.753	.795	.785
Cell 3	0.715	0.728	0.726	.670	.690
Cell 4	0.700	0.718	0.716	.790	.770
Cell 5	0.730	0.740	0.726	.658	.650
Total	3.560	3.630	3.605	3.585	3.575

*Operation unstable, occasional gas surges needed to clear ports.

FIGURE 1



A : CURRENT

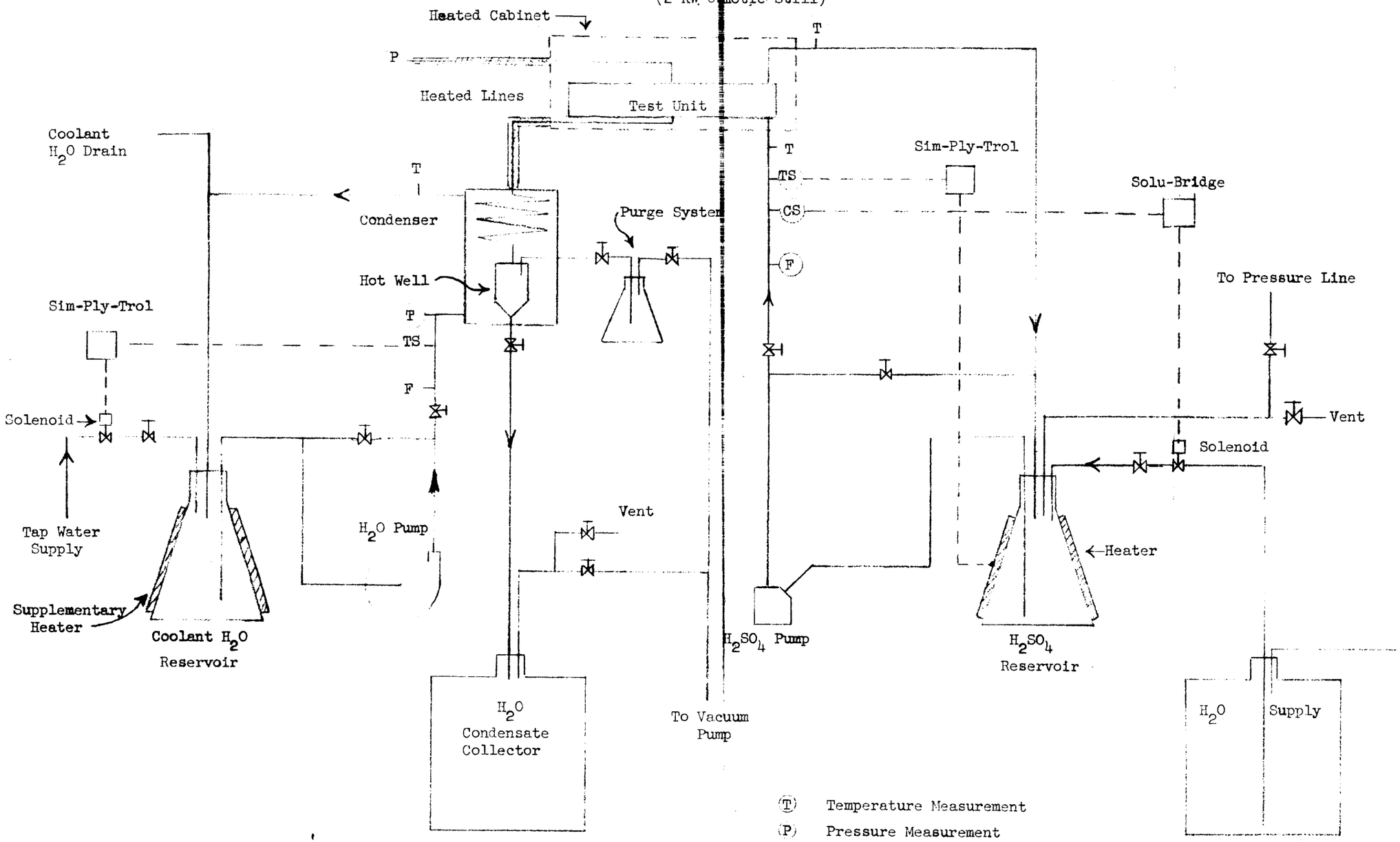
V_T : TOTAL CELL

V_H : HYDROGEN "HALF-CELL"

V_O : OXYGEN "HALF-CELL"

SCHEMATIC OF HOOKUP FOR POLARIZATION MEASUREMENTS.

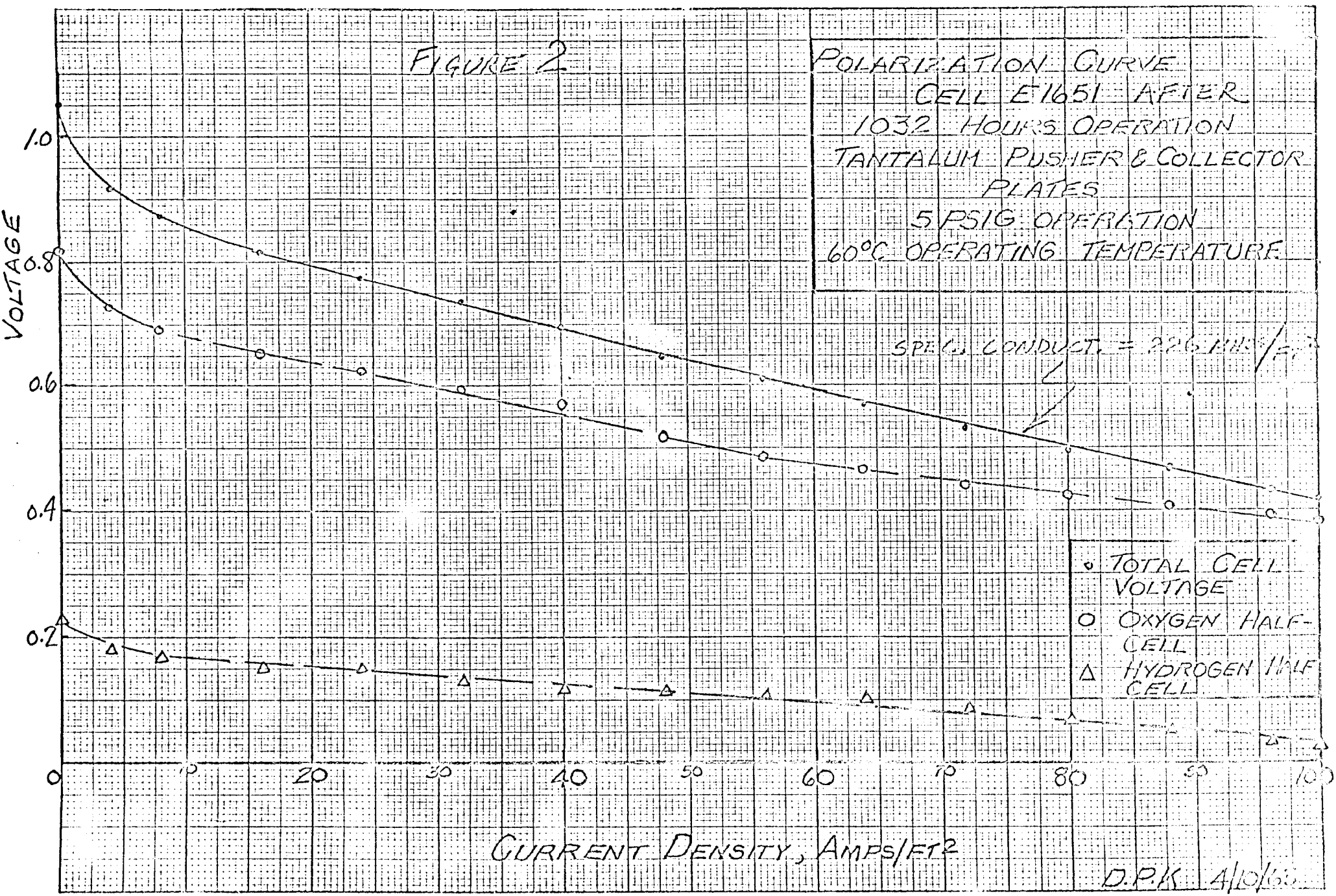
Task II - Test Rig Schematic
(2 KW Osmotic Still)

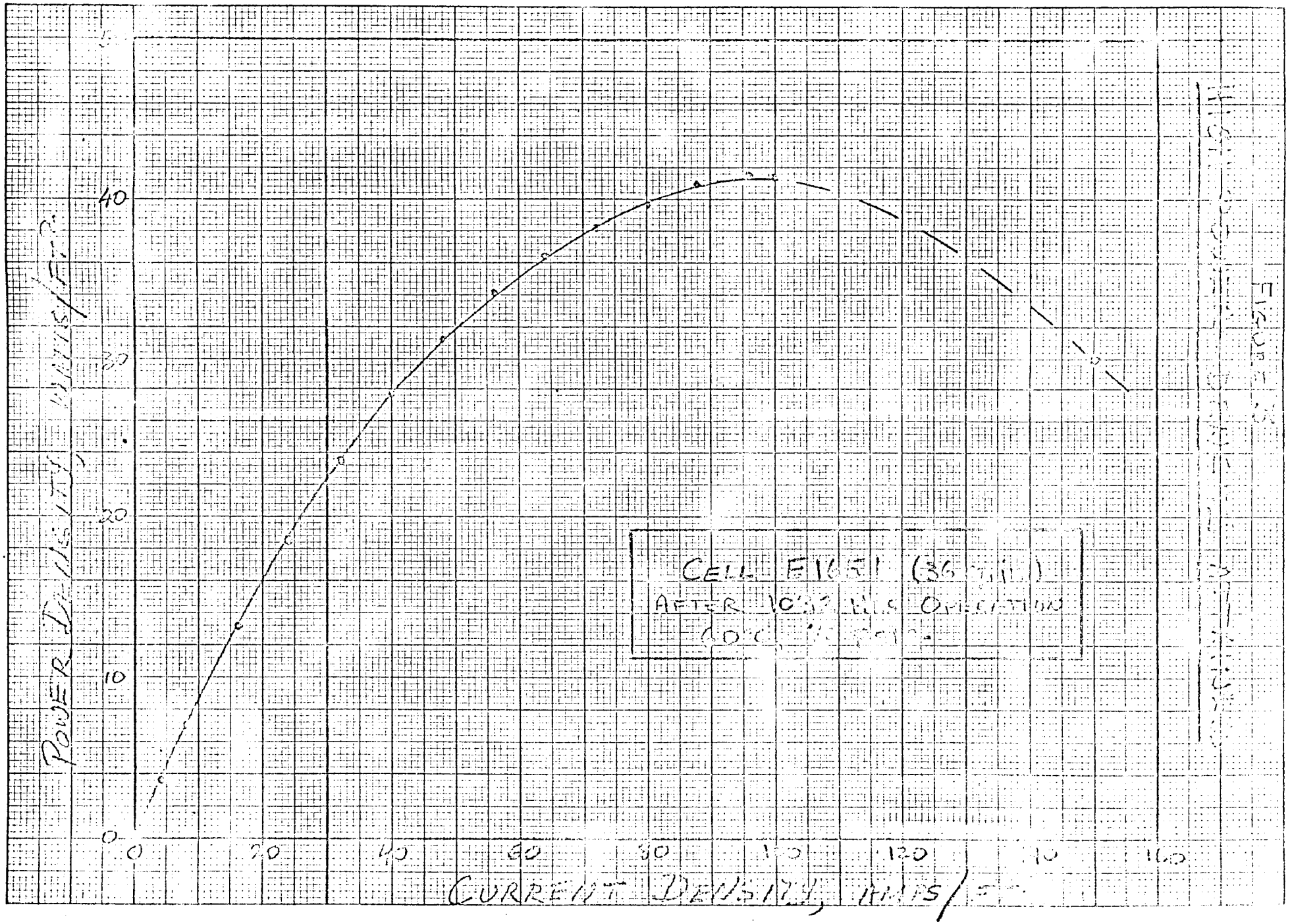


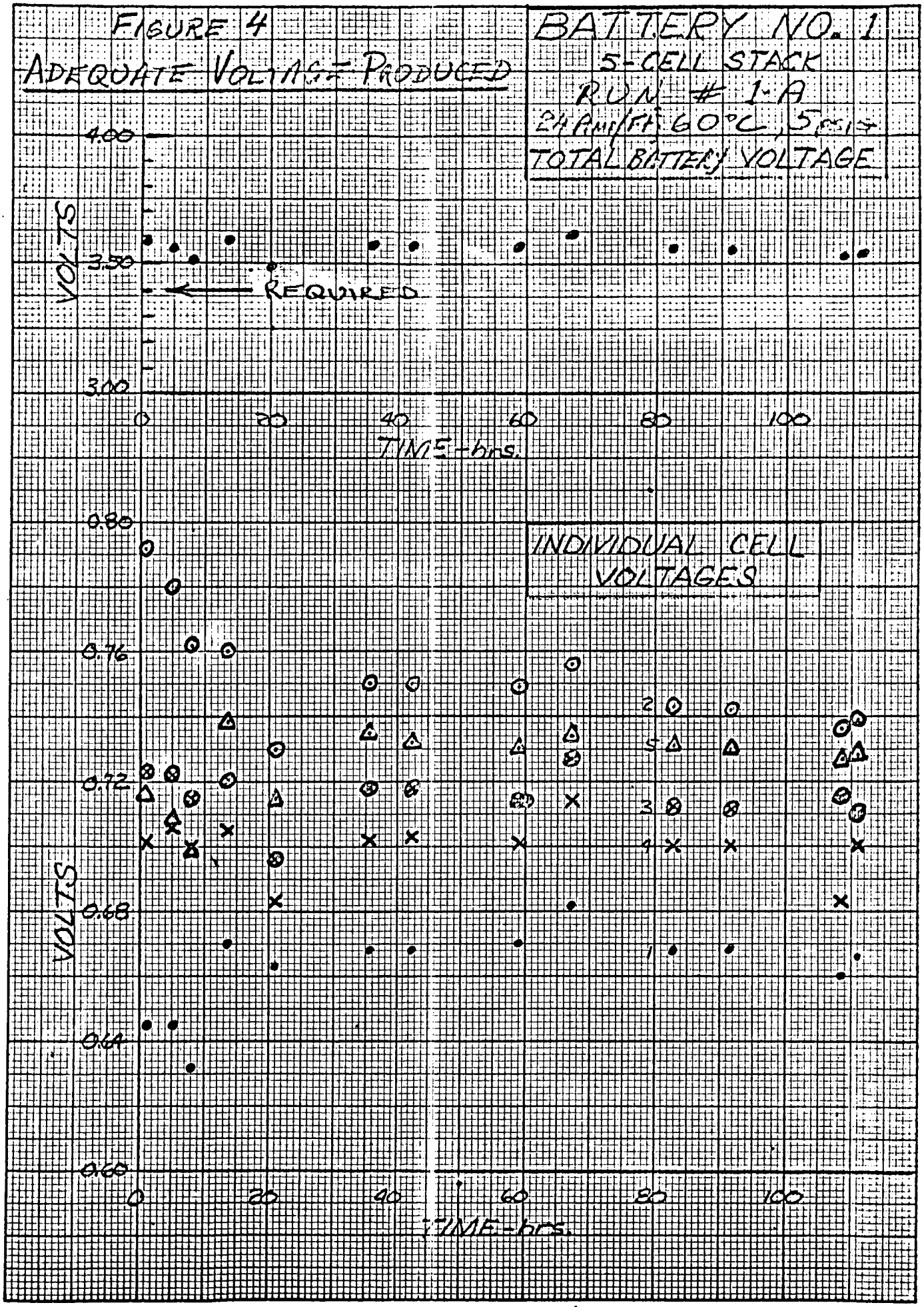
(T) Temperature Measurement
(P) Pressure Measurement
(F) Flow Measurement
(TS) Temperature Sensor
(CS) Concentration Sensor

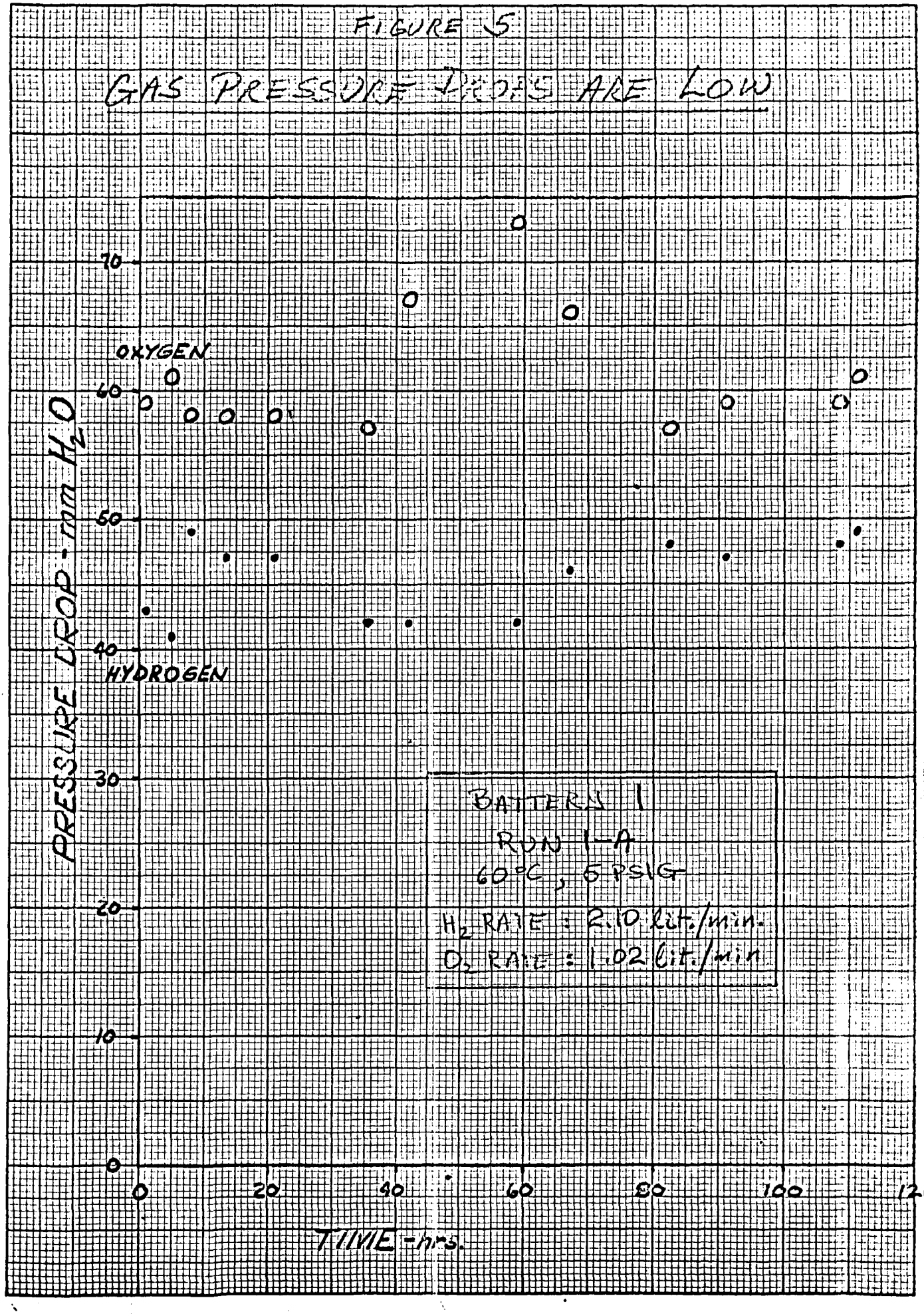
TRW Figure 1A

FIGURE 2.









K&E 20 X 20 TO THE INCH 358-10½ KEUFFEL & ESSER CO. MADE IN U.S.A.

Fig 2

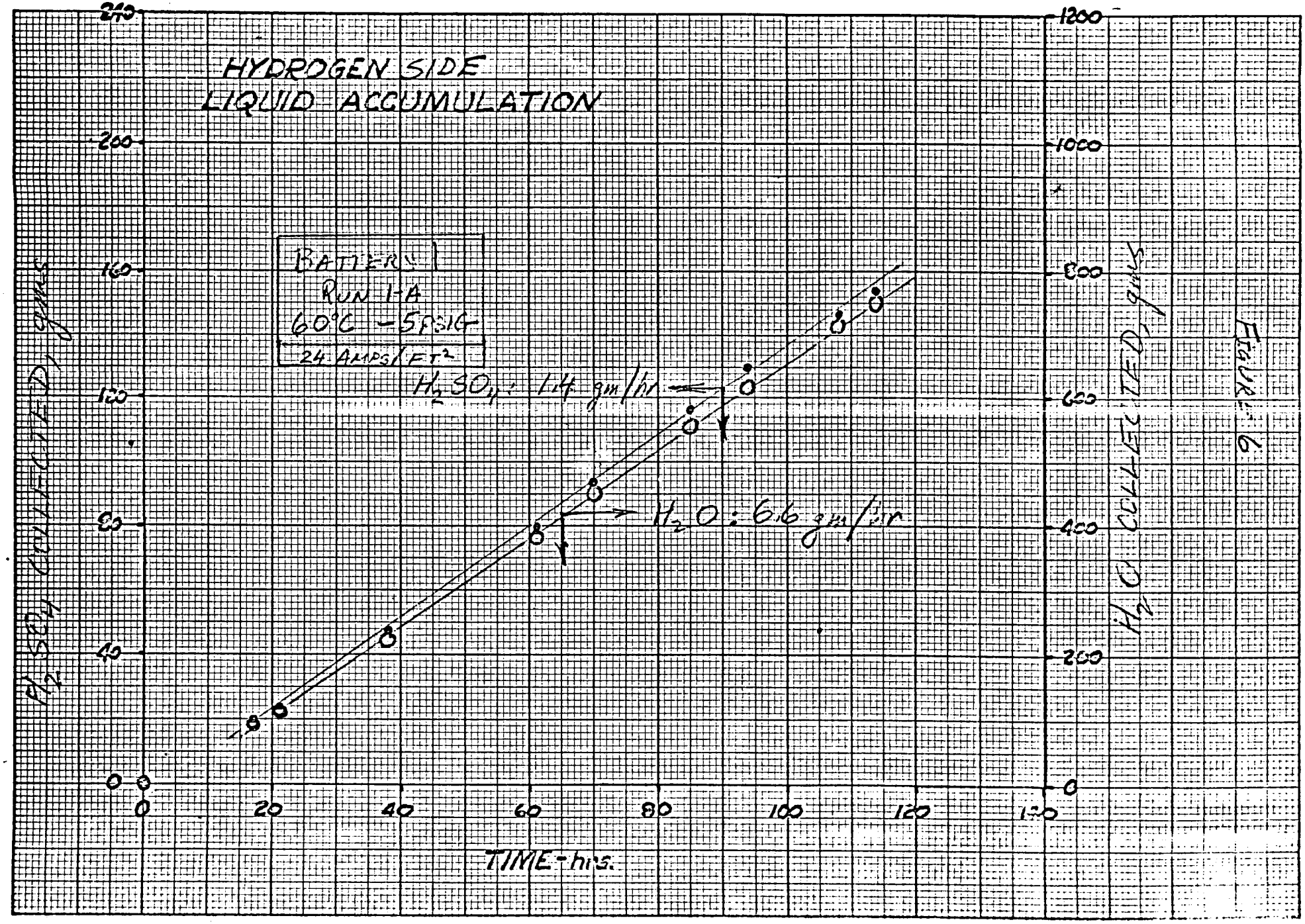


Figure 6

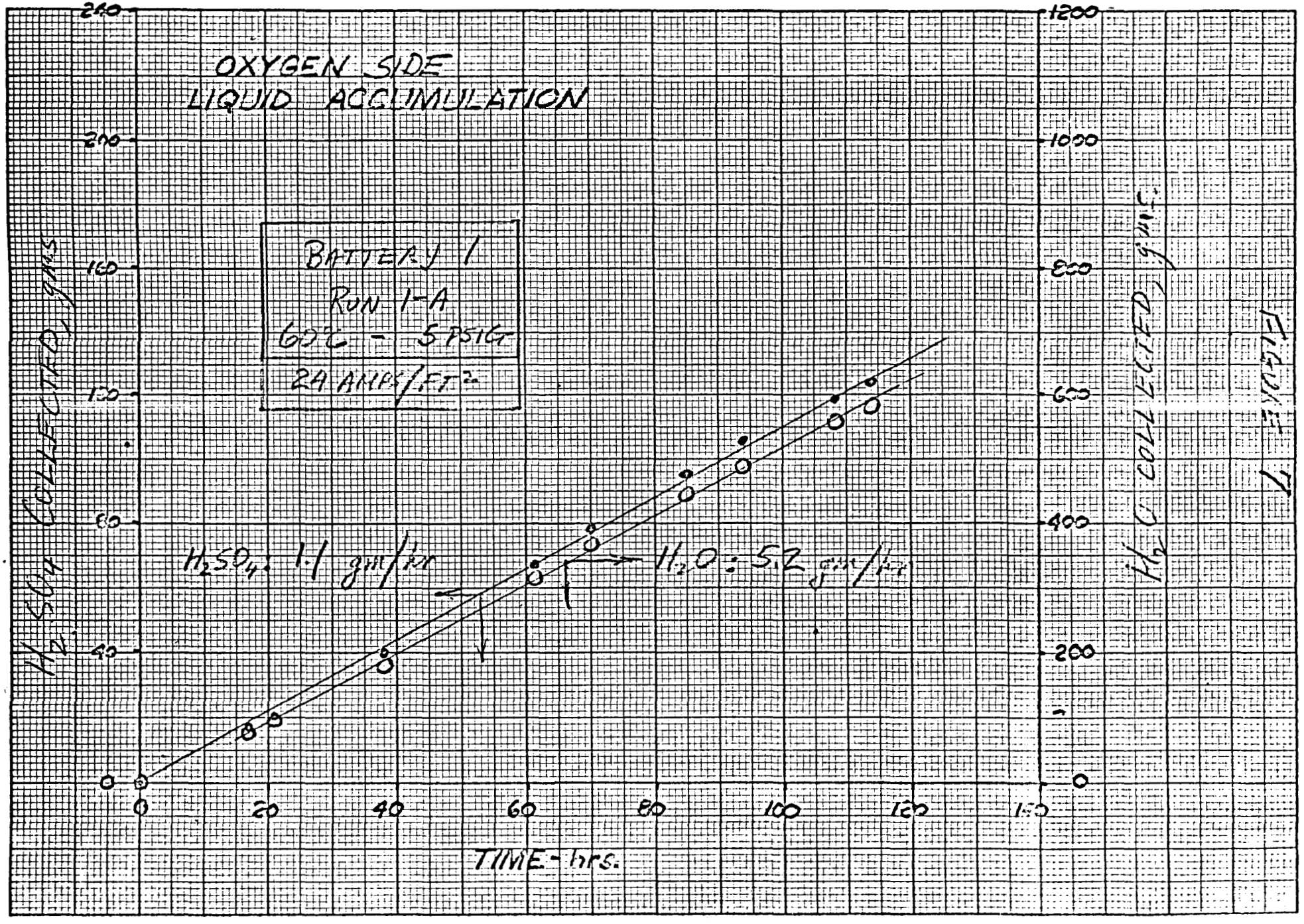


FIG. 3

ELECTROLYTE PRESSURE DROP VS. FLOW: ¼ psi @ 60 ml/min

"PRESSURE DROP THRU BATTERY"

MANOMETER READING, mm OF H2SO4

PSI

RUN 1A
60°C
60 ml 6N H_2SO_4/min

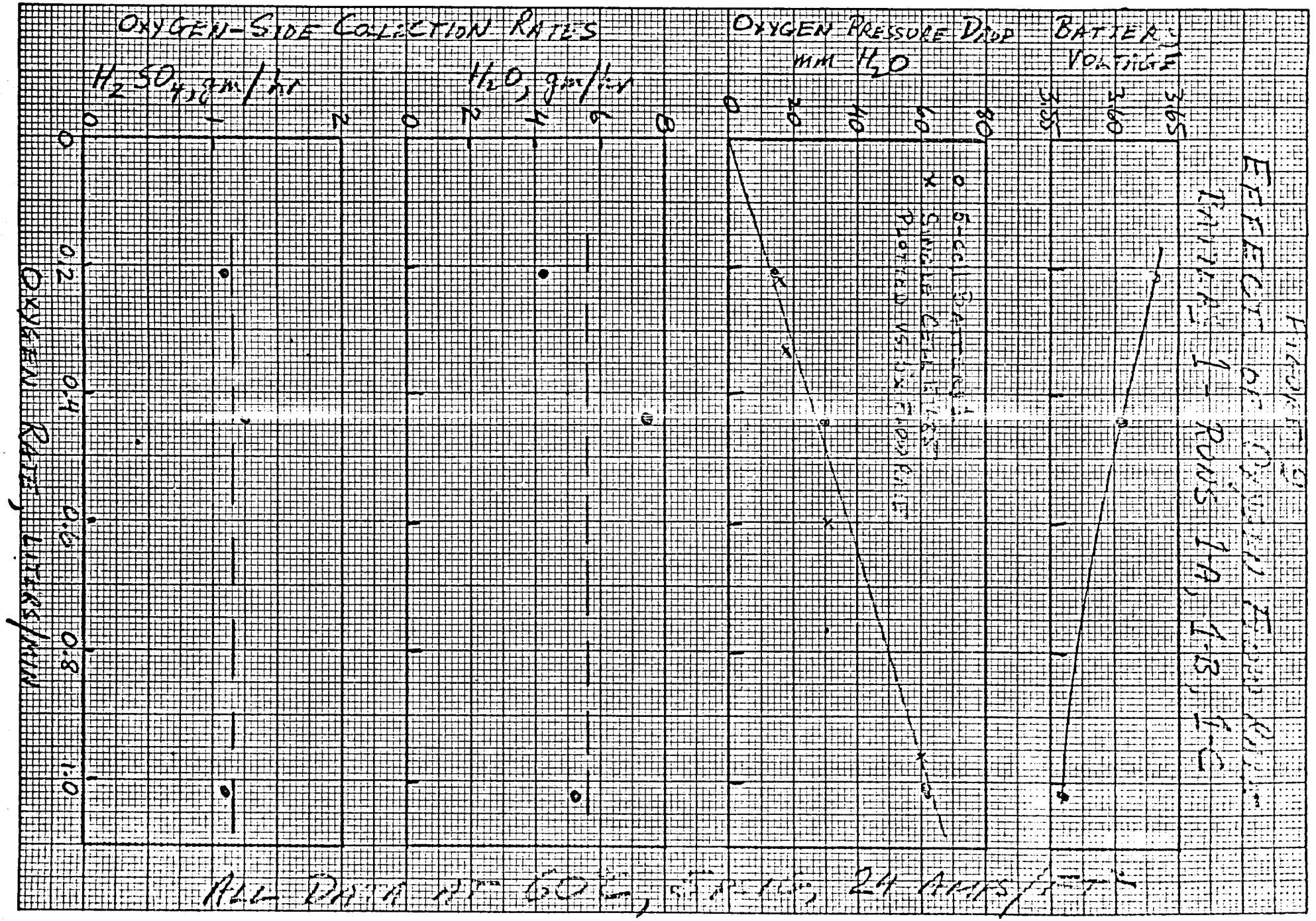
● PUMP PUMPING CONTINUOUSLY
○ PUMP PUMPING (INTERMITTENTLY)
× PUMP NOT PUMPING (")

ELECTROLYTE PRESSURE PSIG

RELAY SET POINTS

TIME — HRS

NOTE: WHEN PUMP IS PUMPING INTERMITTENTLY, THE RESISTANCE-CAPACITANCE CHARACTERISTICS OF THE HYDRAULIC ELECTROLYTE FLOW AND MANOMETER CIRCUITS PREVENT THE MANOMETER FROM READING THE "INSTANTANEOUS" PRESSURE DROP THRU THE BATTERY.



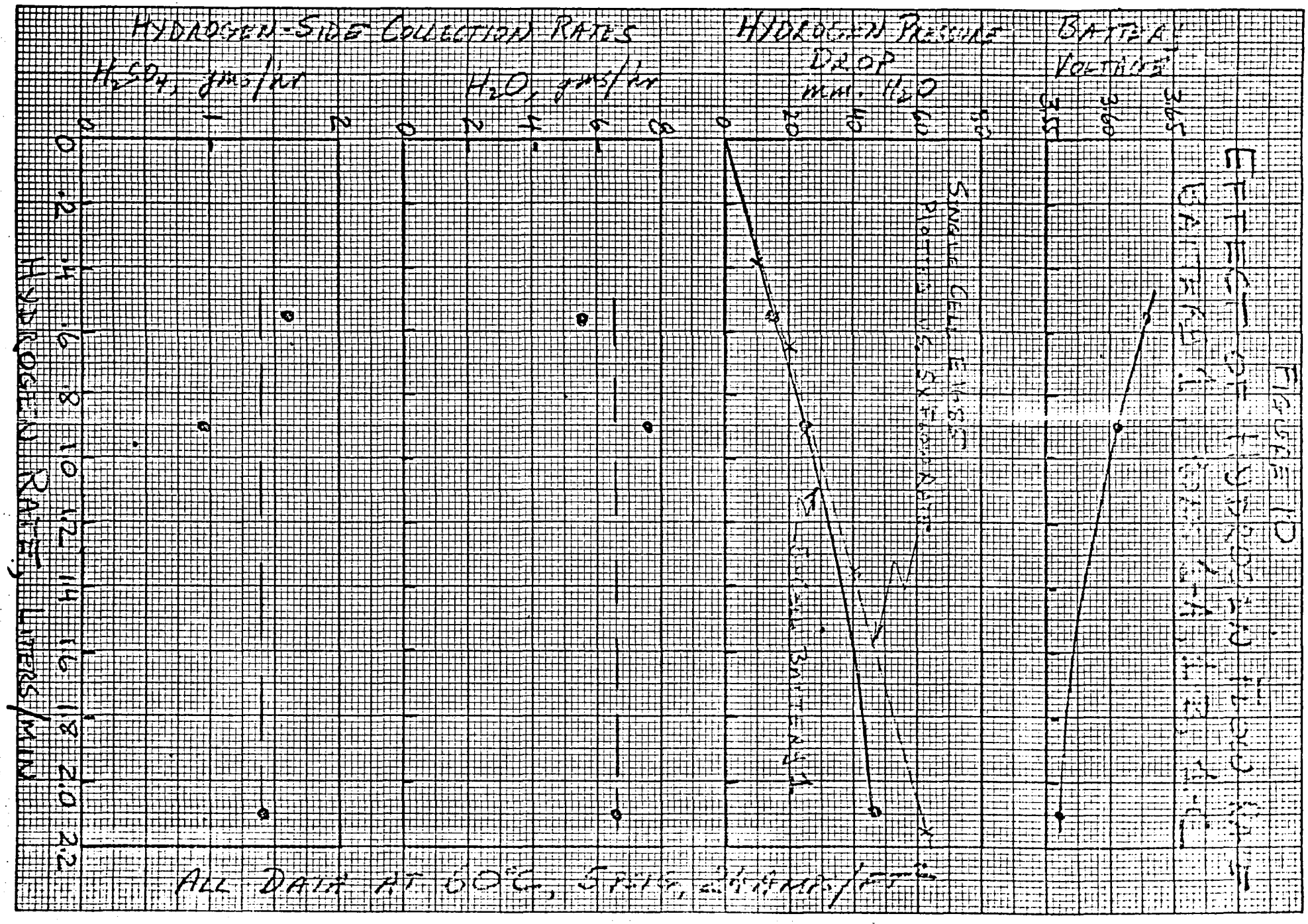
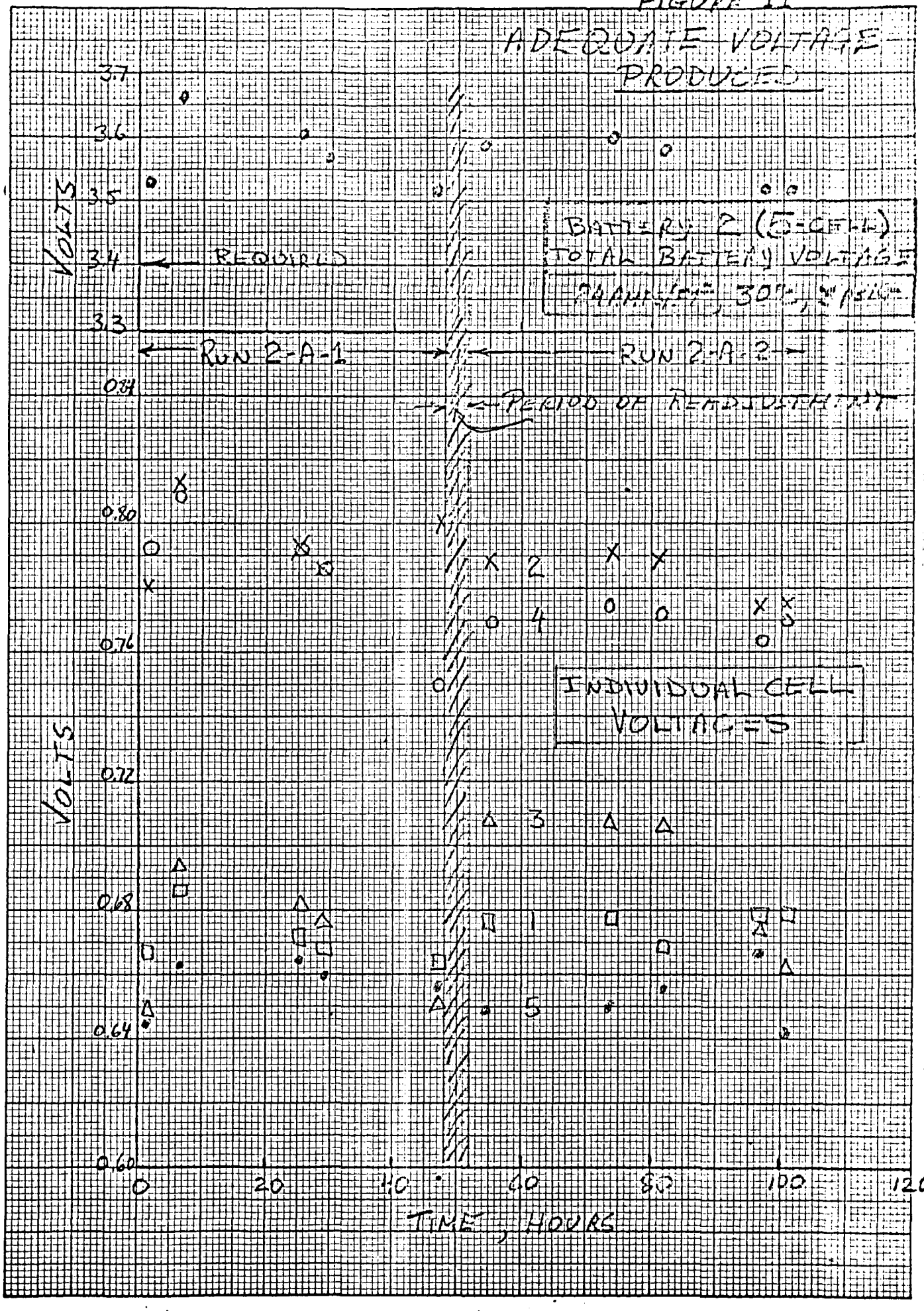
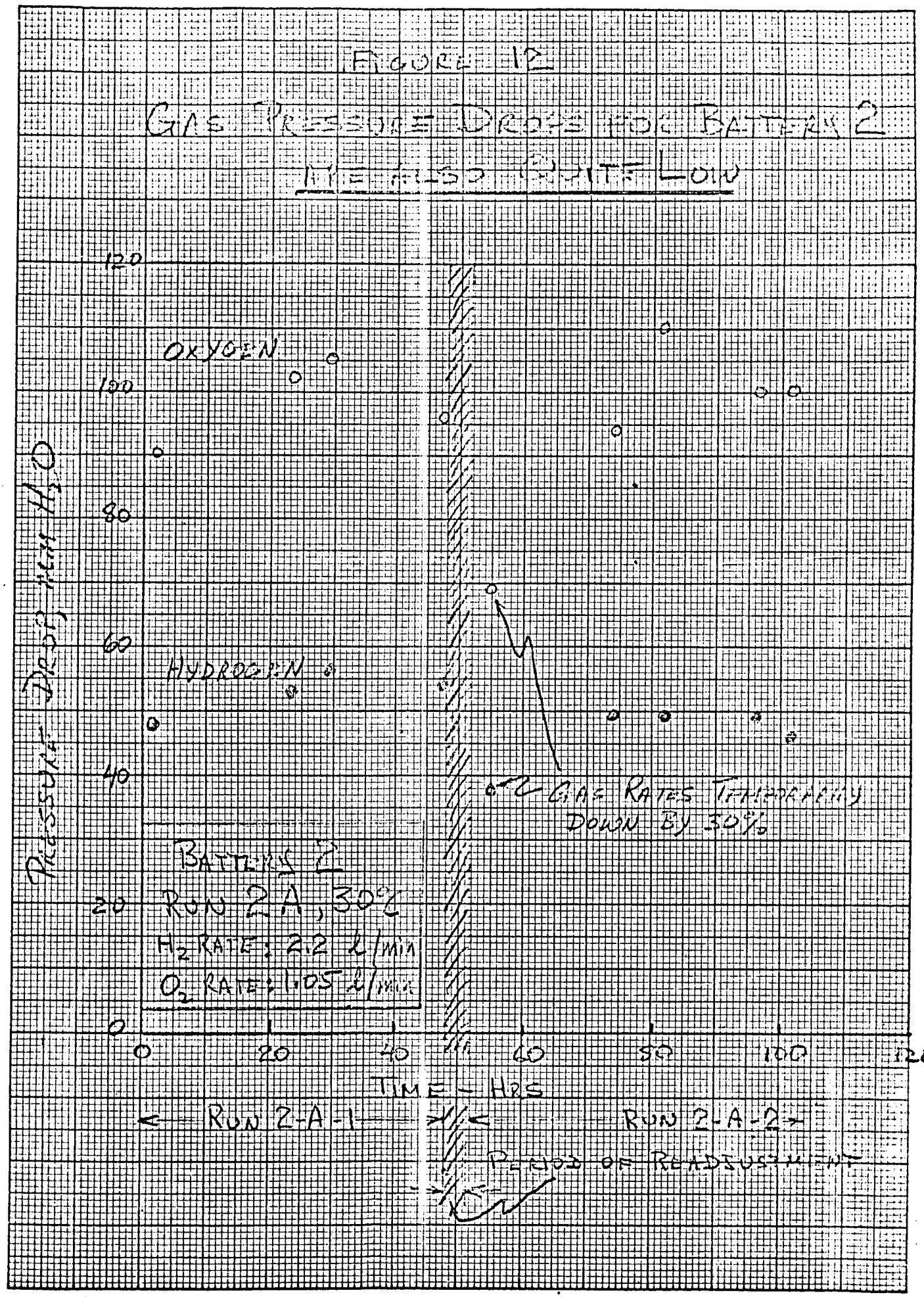


FIGURE 11





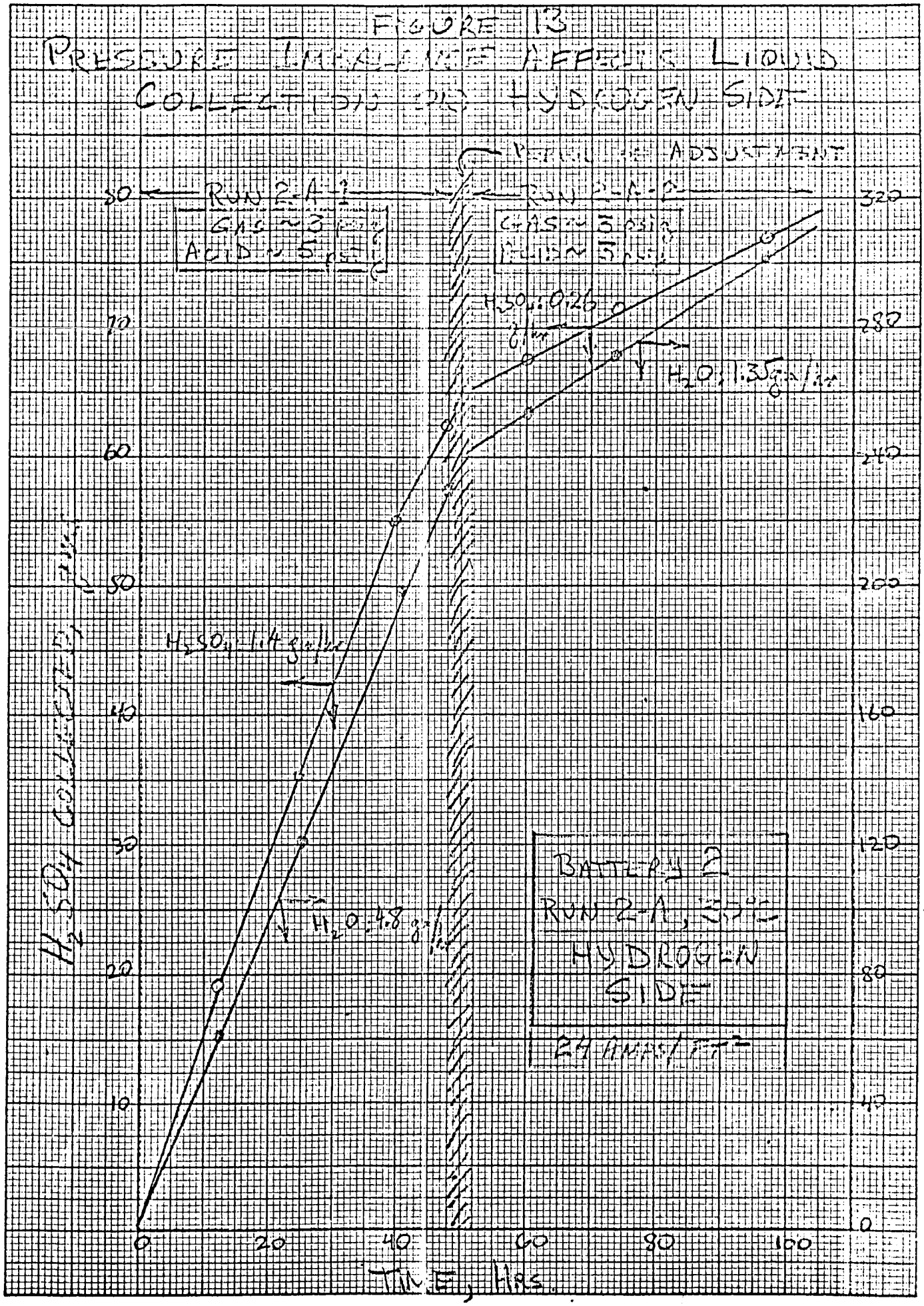


FIGURE 13
PRESSURE INITIAL RATE AFFECTS LIQUID COLLECTION RATE HYDROGEN SIDE
PRESSURE ADJUSTMENT
RUN 2-A
GAS 50 psig
ACID 50 psig
RUN 2-A-2
GAS 50 psig
FLUID 50 psig
H2SO4: 0.26 g/l
H2O: 1.35 g/l
H2SO4: 1.4 g/l
H2O: 4.8 g/l
BATTERY 2
RUN 2-A, 2-A-2
HYDROGEN SIDE
24 HRS TEST
H2SO4 Collected, gms
H2O Collected, gms
TIME, Hrs.
0
20
40
60
80
100
10
20
30
40
50
60
70
80
120
160
200
240
280
320